

AUTOMATION OF A ROUTINE LABORATORY

by R. T. BISHOP and T. D. ALGIE

Introduction

With the recent expansion of the sugar industry in South Africa a substantial increase in the number of soil and leaf samples submitted to the Fertiliser Advisory Service of the South African Sugar Association was predicted. To maintain the efficiency of this laboratory it was necessary to revise many of the existing analytical techniques. Procedures used for normal quantitative analysis of soils and leaves involve preparation of the sample, extraction of elements from the sample, filtering and dilution of the

extract and determination of the concentrations of elements present. Some suggestions to facilitate each of these operations are presented below.

1. Grinding of Soil Samples

A simple soil grinder developed at the Soils Testing Laboratory at Raleigh by Dr. P. D. Reid (Director, Soils Testing Division, North Carolina, Department of Agriculture) can be easily constructed. The plans of a modified version of this grinder being tested at Mount Edgecombe are presented in Figure 1.

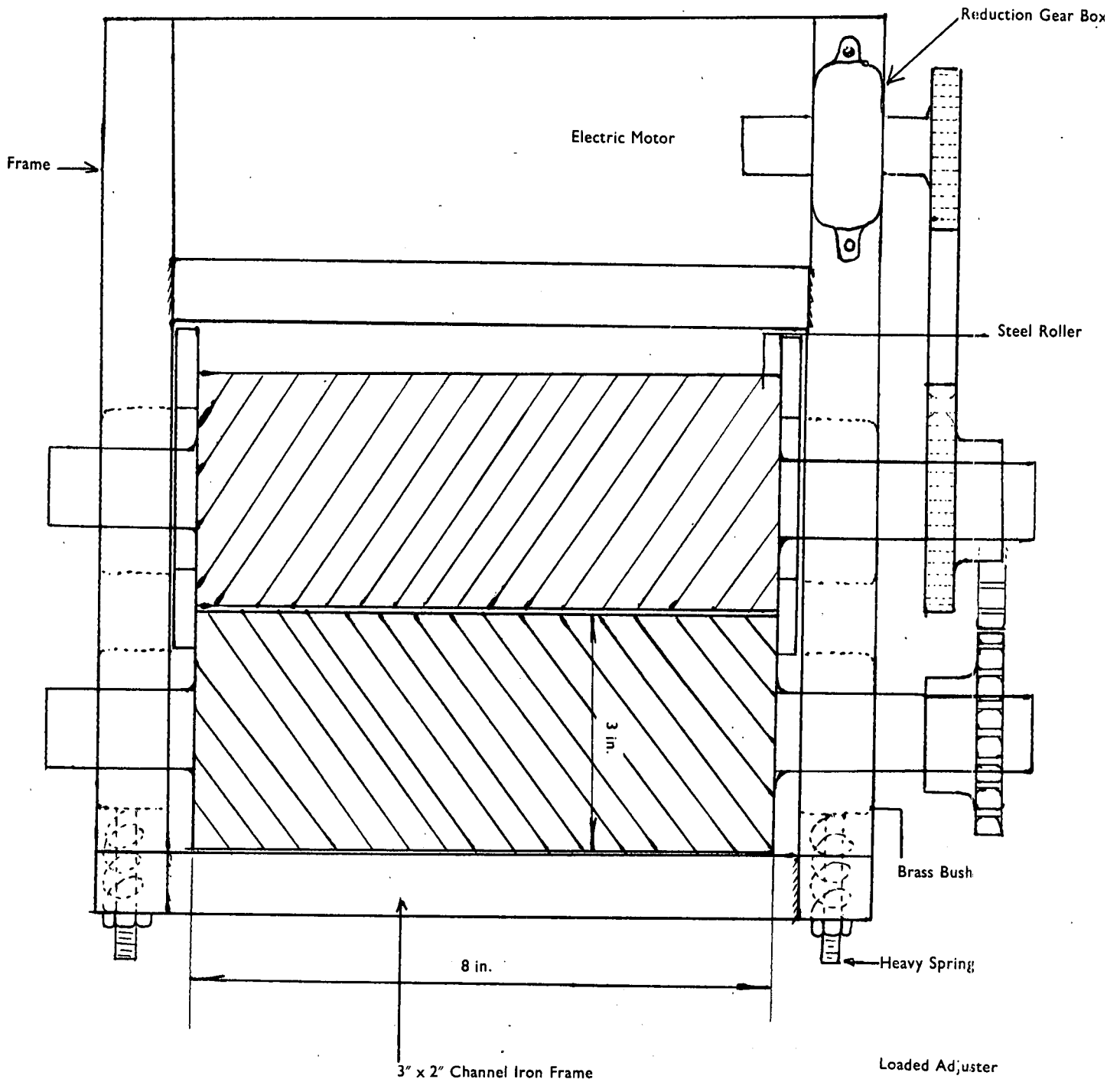


FIGURE 1. Plan of a simple soil grinder.

The two case-hardened steel rollers which rotate in opposite directions are spring loaded at 2 mm. apart and are driven by a 0.33 h.p. electric motor. The rollers are swept clean by the two brushes mounted below and parallel to them. After passing through the rollers the soil is funnelled into a receiving vessel. Sieving of the soil is a separate operation. To eliminate cleaning of the bench after grinding and sieving of each sample, these operations are carried out over a wire frame mounted above a funnel and dust bin.

2. Weighing

Normal quantitative chemical analyses involve the addition of a known volume of solution to a known weight of material. Provided the ratio of solution to material is kept constant it is not necessary to know the exact weights and volumes used. A "ratio-balance" employing this principle was observed at Oosterbeek (Laboratory of Soil and Crop testing, Oosterbeek Holland) and an instrument designed at the Experiment Station of the South African Sugar Association to achieve the same end is illustrated in Plate 1.

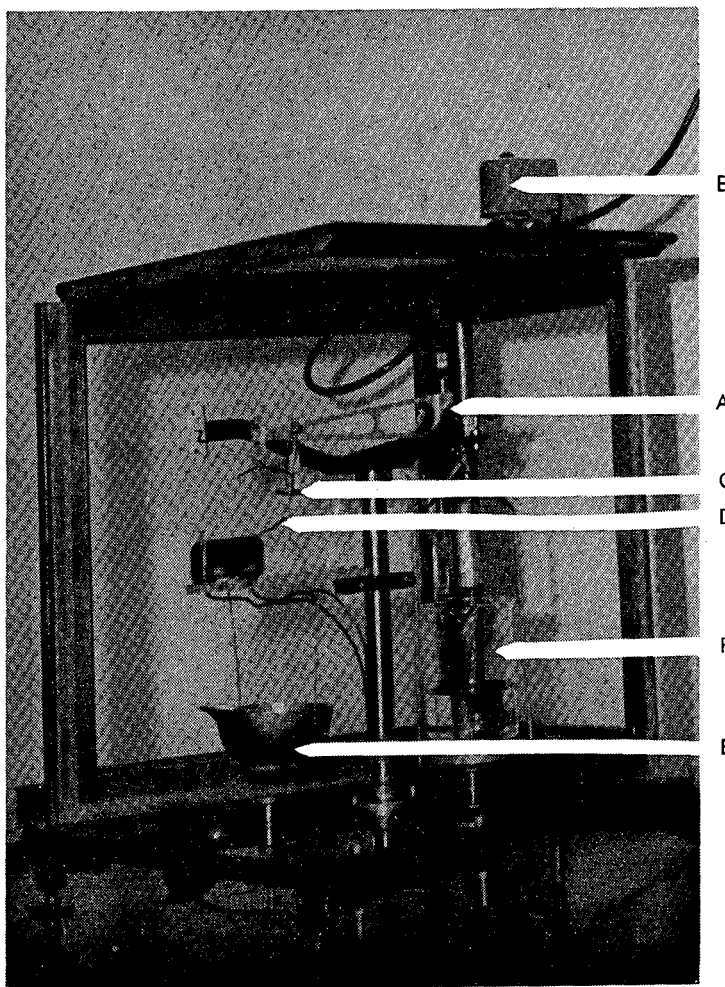


PLATE 1. Balance used at Mount Edgecombe for providing a solution to soil ratio of 10:1.

By making the ratio of the distances from the ends of the balance arm to the knife edge (A) 10:1, a weight ratio of the same order is possible. In the extraction of exchangeable cations (normally exactly 10g. of soil are weighed out and leached with exactly 100 ml. of N. ammonium acetate) a spoonful of soil which weighs approximately 10g. is added to the tared container (B). The addition causes the balance arm supporting the soil to swing down and contact is made between the metal disc (C), which is insulated from the rest of the balance arm, and the micro-switch (D). This contact completes an electrical circuit which opens the solenoid valve (E). Ammonium acetate is injected into the tared beaker (F)

until the weight ratio of solution to soil reaches 10:1. At this point the balance arm swings upward, breaking the electrical circuit and closing the solenoid valve.

3. Handling of Glassware

Where a number of samples are to be subjected to the same treatment, the speed of each operation e.g. pouring, filtering, washing etc. can be greatly increased by fixing the required glassware permanently in 18 gauge aluminium racks. In this way ten or more samples can be handled simultaneously. The tray sizes adopted at the Experiment Station are 26 in. long by 3 in. wide (height measurements will vary

with the glassware used). Two such racks, one used for the extraction of acid soluble P from soils and the other for receiving the filtered solution, are included in Plates 2 and 4 respectively.

4. Extraction

To determine the concentrations of elements in biological material it is generally necessary to bring

them into solution. This procedure normally requires shaking of some kind and two shakers which are operating satisfactorily are shown in Plates 2 and 3.

(a) End over end shaker

The shaker presented in Plate 2 can hold 110 vessels and the wooden drum is 27 in. long. Other measurements are given in Figure 2.

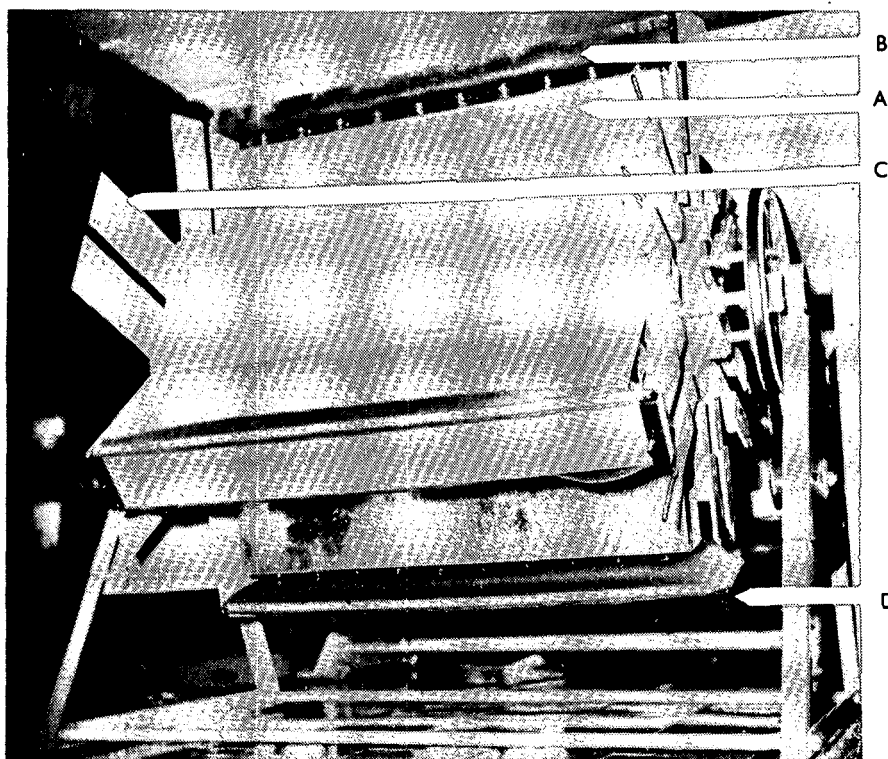


PLATE 2. End over end shaker capable of holding 110 vessels.

The aluminium rack (A) is held in position by the stopper (B). This stopper consists of a strip of 2 in. thick foam plastic covered by rubber which is tacked to a half-inch thick wooden board. Bolts (1 in. x 4 BA) fixed to a hinge on the end of the board are slipped through holes in the bracket (C). The other end of the stopper is screwed down (D) effectively sealing the mouths of all 10 containers. The stoppers are removed and washed between extractions.

(b) Vertical shaker

The vertical shaker used at Mount Edgecombe was designed by R. A. G. Rawson Rothampstead Experimental Station, Harpenden, England, formerly of S.A.S.A. Experiment Station) and makes use of the block of a motor car engine in which pistons 1 and 4 fired simultaneously. (See Plate 3).

The engine block (A) is mounted on concrete supports and the piston rods of cylinders 1 and 4 (B) have been extended upwards above the

level of the bench. The frame of the shaker (C) is secured to these rods. A flywheel is fixed to the front of the crankshaft and a 0.33 h.p. electric motor (D) provides the necessary drive.

5. Removing Aliquots

The possibility of using a vacuum-compressed air system for removing equal aliquots simultaneously from a number of different solutions was suggested by Dr. Reid. A piece of equipment utilising this idea has been constructed at Mount Edgecombe and is in daily operation. (See Plate 4).

When the system is subjected to a vacuum with the tips of the pipettes in solution a constant suction is maintained when incoming air is drawn down the glass tube (A), which is immersed in the container of water (B), to compensate for the air removed. By raising or lowering the depth of this tube in the water the degree of vacuum in the whole system can be altered. To remove exactly the same aliquots in all ten pipettes it is essential that the distances between their tips and graduation marks be the same.

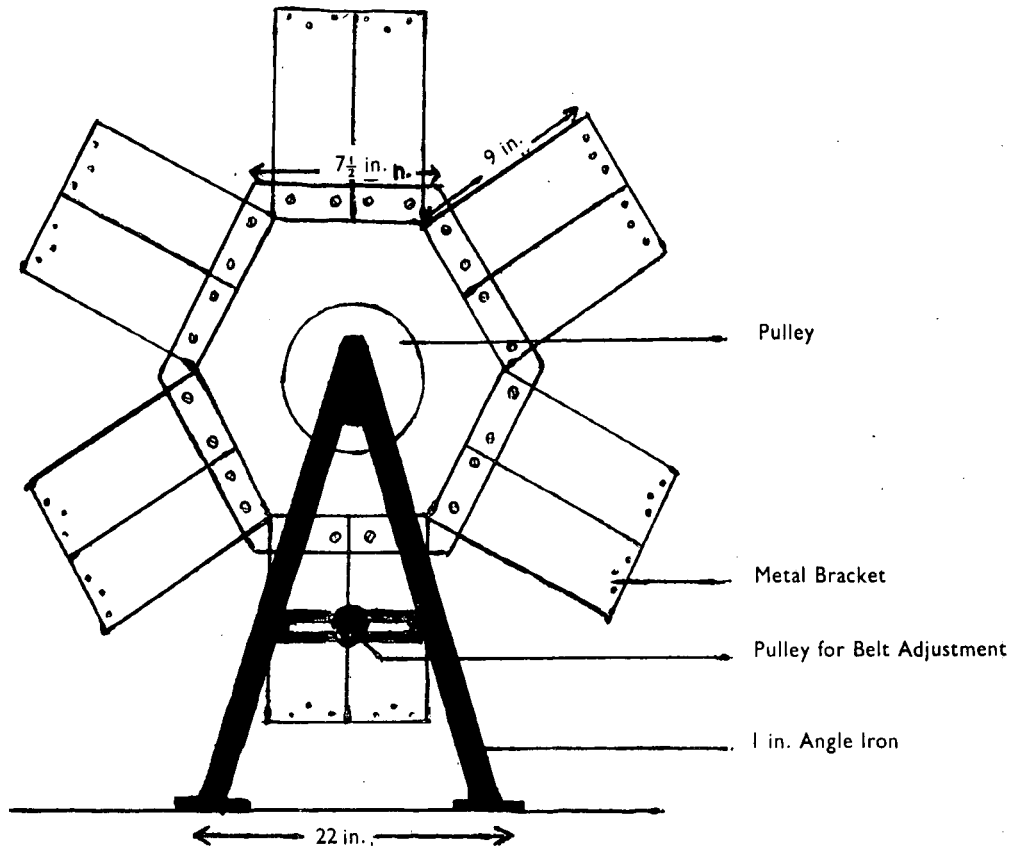


FIGURE 2. Front view of end over end shaker.

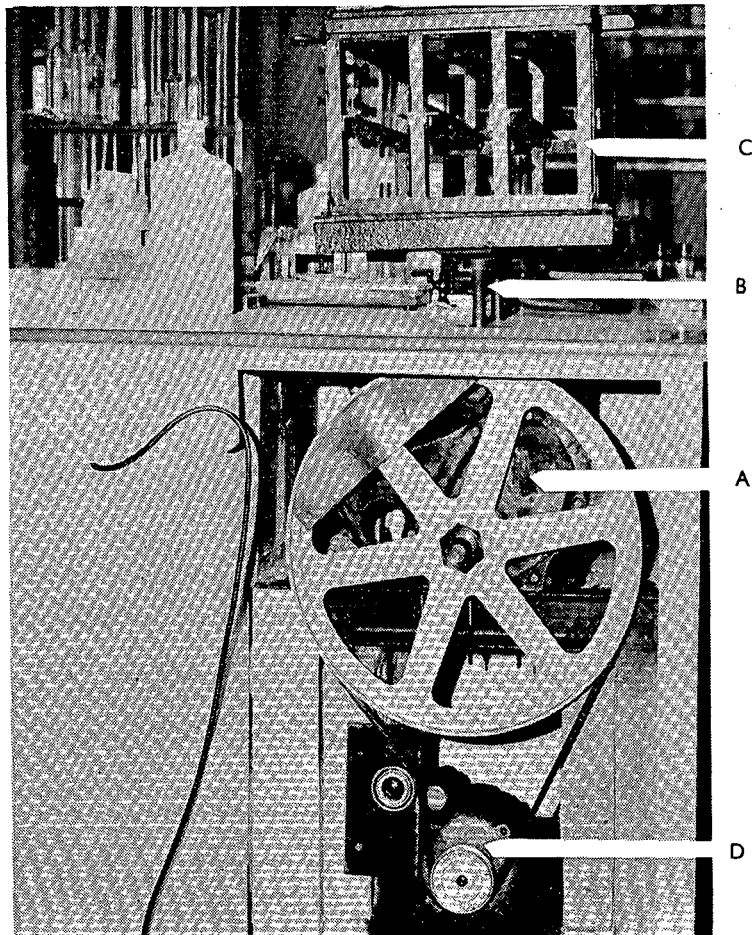


PLATE 3. Vertical shaker modified from the block of a petrol engine.

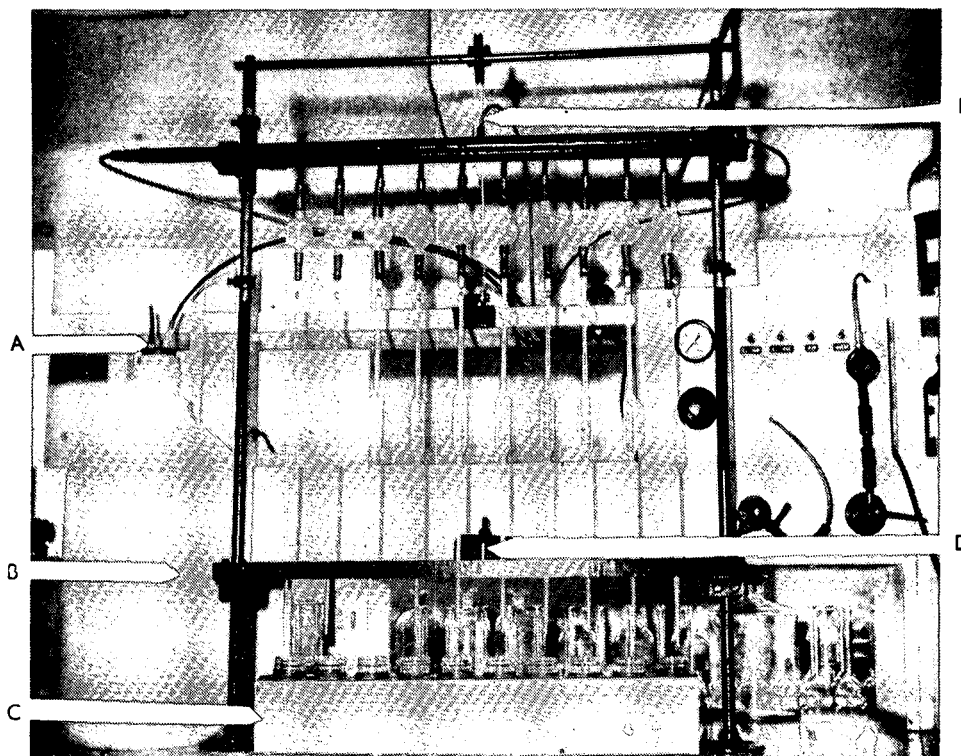


PLATE 4. Apparatus used for removing 25 ml. aliquots simultaneously from 10 solutions.

The steps involved in the operation of the apparatus are:

- (a) The rack of solutions to be sampled (C) is lined up under the pipettes. The pipettes are lowered manually into the solutions and the high vacuum switch is turned on.
- (b) When the pipettes are approximately 75 per cent full (after about 2 seconds) the high vacuum switch is released and the low vacuum switch is depressed.
- (c) When the levels of solutions in the pipettes are maintained at the predetermined heights the pipettes are lifted out of the solutions. The counter weight (D) permits the pipettes to be raised easily and smoothly.
- (d) A rack of receiving vessels is inserted under the pipettes and the compressed air switch (which isolates the water cylinder from the rest of the system and exposes the pipettes to a compressed air supply) is depressed thus ejecting the solutions into the receiving vessels.
- (e) The receiving vessels are removed and the water supply is turned on releasing a supply of deionised water (E) into each pipette.

The circuit diagram of the electrical system of the apparatus is given in Figure 3.

6. Measurement

Two techniques commonly used in the laboratory for measuring concentrations of elements are colorimetry and flame photometry.

Tubes for the colorimeter are expensive and the general procedure is to use only one tube which must be rinsed and then refilled before the colour intensity of each solution can be measured. Although modern instruments permit the filling and emptying of the colorimeter tube to be carried out automatically, most laboratories still have the older type. By using thin walled glass specimen tubes in place of the colorimeter tubes results of an accuracy acceptable for fertiliser advisory work have been obtained. Colour development is carried out directly in these tubes which are then inserted into the colorimeter.

Filter type flame photometers are commonly used for K, Ca and Na determinations. For the determination of concentrations of each of these elements in the same solution a different light filter must be inserted into the instrument. This means that for K, Ca and Na determinations each solution must be atomised three times. A modification of this instrument which permits the three elements to be determined during a single atomisation is illustrated in Plate 5.

The unit consisting of focusing lens, filter holder and selenium cell found in the conventional instrument (A) has been reproduced on the opposite side of the flame (B). The new guides for the filter have been extended (C) to accommodate two filters one mounted above the other. The Ca filter is placed above the K filter, and to increase the sensitivity of the instrument to the former element, the reflector (D) is fixed opposite to it. The Na filter is inserted in the normal filter holder.

When operating the instrument the Ca filter and reflector are pushed down into line with the selenium

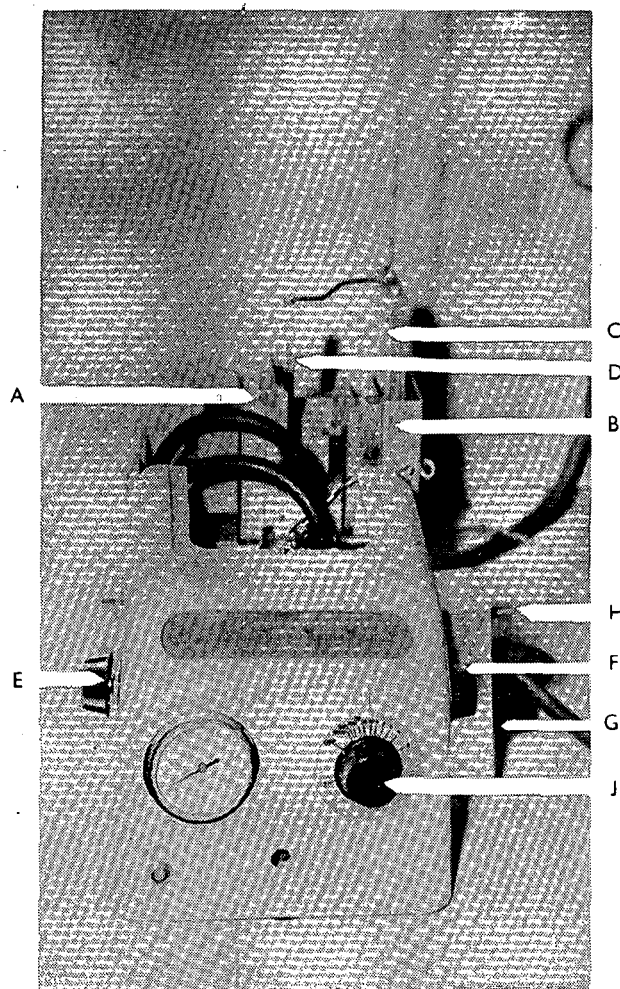


PLATE 5 Modified filter type flame photometer used for determining K, Ca and Na concentrations during a single atomisation.

cell. Knob (E) is set on position 1 and the blank is set on O using knob (F). The top standard is set on 100 with knob (G). The Ca filter is pulled upwards and the K filter automatically lines up with the cells. Knob (E) is set on position 2 and the top standard is set on 100 using knob (H) and the reading of the blank is noted. Knob (E) is set on position 3 and knob (J) is used to set the top Na standard on 100. The blank reading is again noted. The unknown solutions are then atomised and by altering the setting of knob (E) the concentrations of Ca, K and Na are reflected on the common galvanometer. The filters for Ca and K must be changed each time and the blank readings for K and Na subtracted from the observed results.

A circuit diagram of this instrument is presented in Figure 4.

Summary

Descriptions, together with diagrams and/or plates, are given of apparatus suitable for (1) grinding soils, (2) measuring out solution to solid ratios of 10:1, (3) handling 10 glass containers simultaneously, (4) shaking either end over end or vertically, (5) removing equal aliquots simultaneously from 10 different solutions and (6) measuring the concentrations of K, Ca and Na during a single atomisation of the solution into a filter type flame photometer.

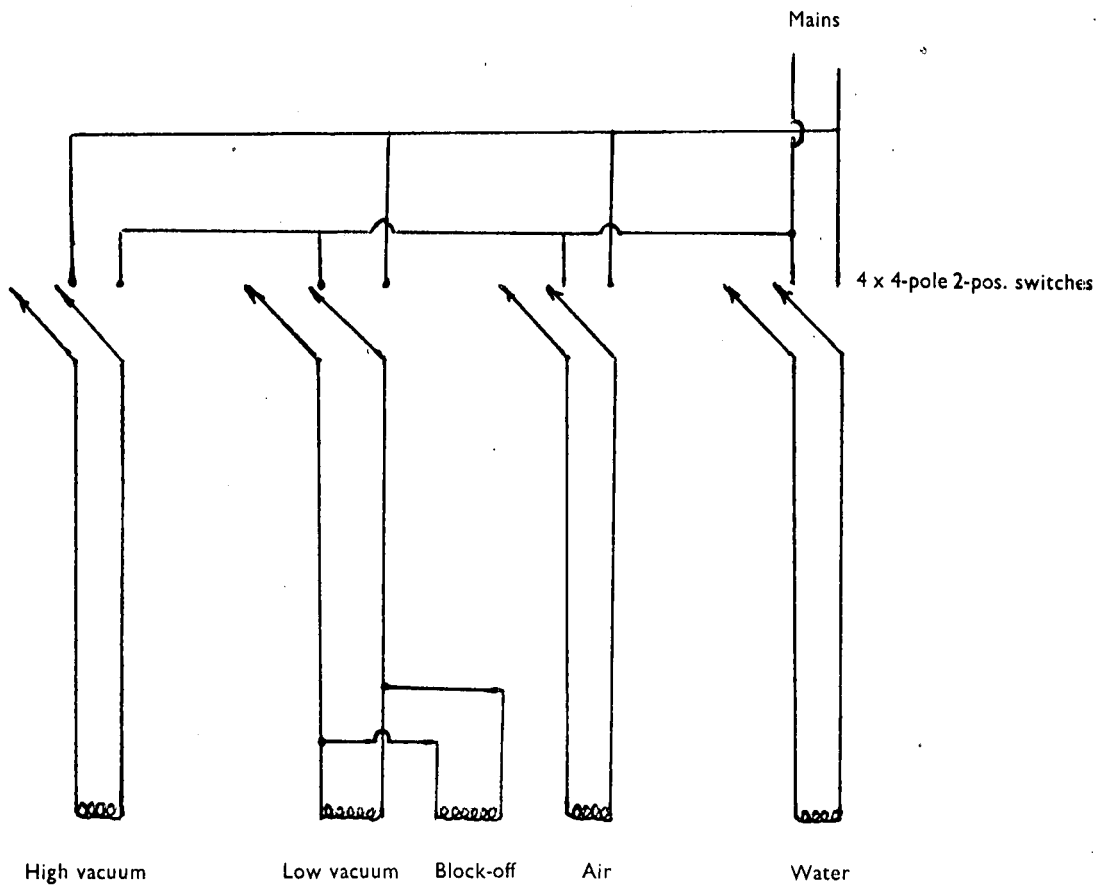


FIGURE 3. Circuit diagram of the electrical system of the apparatus used for removing equal aliquots.

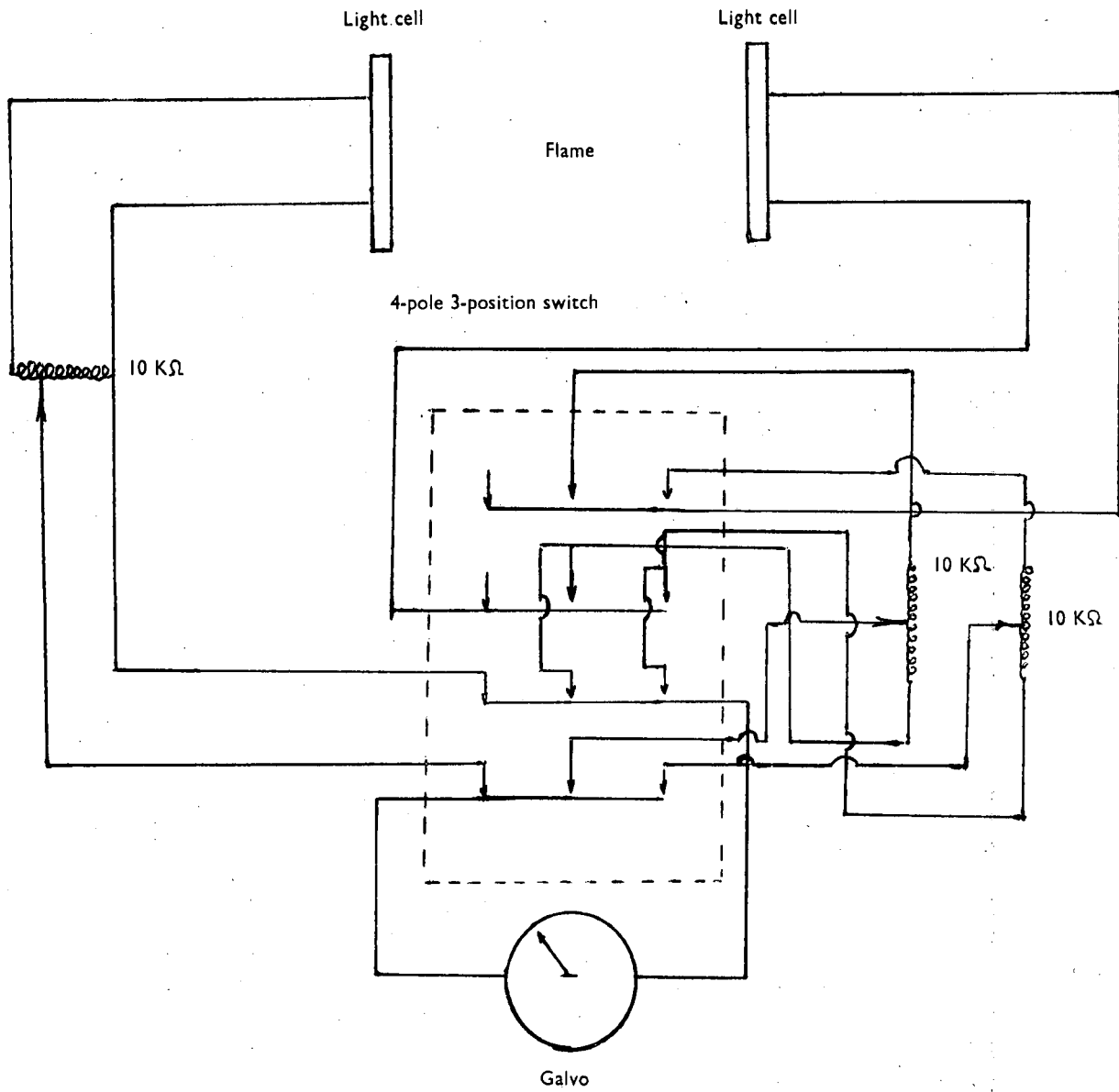


FIGURE 4. Circuit diagram of modified filter type flame photometer.